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(54) An apparatus and method for controlling the cross-linking of elastomers in a mould.

(57) Apparatus for checking or controlling cross-linking in products made of elastomeric material which is the object of the present invention, comprises a mould of a fixed volume provided with at least one pressure sensor able to detect pressure variations in the mould during cross-linking in order to obtain data referring to the progress of the cross-linking in the case of laboratory applications, while, should the apparatus be used in manufacturing, it can provide an automatic control of the duration of the various stages of the manufacturing cycle by means of microprocessor controlled equipment to which such sensors are connected.

The operating method according to the present invention comprises measuring the peak pressure reached in the mould, detecting a decrease in its gradient as time passes, subsequently detecting an increase in the gradient and determining the time required to obtain a variation of a pre-established order, this data being used to obtain information on how cross-linking is progressing and for automatically controlling the manufacturing equipment.

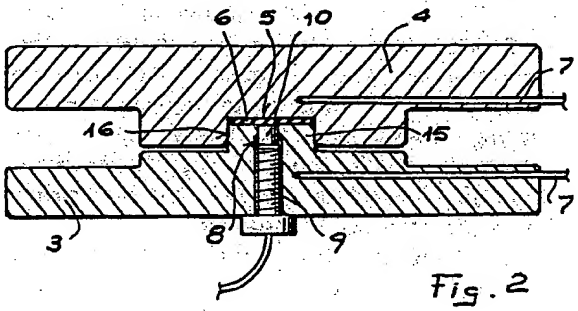

Fig. 2

EP 0 279 081 A1

AN APPARATUS AND METHOD FOR CONTROLLING THE CROSS-LINKING OF ELASTOMERS IN A MOULD

The present invention relates to a method and an apparatus for checking or controlling the cross-linking of products made of moulded elastomeric material, which enables both the laboratory assessment of the typical parameters of cross-linking and the control of the period during which cross-linking occurs in actual production to be carried out.

Products of elastomer material, such as tyres and the like, are usually formed within a mould, in which they are kept until the completion of the cross-linking process of the material which is brought from an initial fluid or paste-like state into the solid state characteristic of the finished product.

The cross-linking, which consists of the formation of transversal links between the chains of the polymer forming the elastomer, takes place in a time dependent upon a number of factors related both to the composition of the mixture and the agents used for the cross-linking and also to external factors, such as pressure and temperature; and it is therefore essential experimentally to establish how the mixture under examination progresses as time goes by in order to be able to supply precise operating data for a successful moulding operation.

It is in fact essential for the item obtained to be kept inside the mould until cross-linkage is completed or at least until it has reached a certain stage, enabling the item to keep the required shape, without however unduly prolonging its stay in the mould, thus avoiding the hampering of productivity and preventing excessive cross-linking which would cause an alteration in the properties of the product.

To this aim and according to a known technique, laboratory tests are carried out on a sample of the mixture out of which the item has to be made, in order to establish the required length of setting time under specific operating conditions and thereby enabling the moulding apparatus to be adjusted accordingly.

To this aim, vulcanisation testers having oscillating rotors are used, among other means, according to agreed standards, the rotor being inserted in a chamber containing the mixture undergoing cross-linking and being kept in oscillating motion within it, and the variation with time of the torque required to maintain the oscillation amplitude constant being measured.

A considerable increase in the measured torque provide an indication that cross-linking is taking place, such cross-linking being completed when the measured torque again becomes constant or varies only slowly, having higher values than those of the initial torque.

The data referring to the variations of the torque throughout the time during which cross-linking is taking place enables the time required to be established, under specific conditions, for cross-linking to be carried out in the moulding to the required degree.

This method, however, is onerous as it requires a vulcanisation tester equipped with an oscillating rotor, which is a complex instrument with a delicate structure; moreover the condition under which th mixture being tested adheres to the walls of th chamber and to the rotor is critical, and finally production conditions must be kept constant because, should they vary, the data obtained during the tests would lose their value.

Therefore there is a need for the provision of a process and of an apparatus for measuring th progress of cross-linking which is easy to work and which also makes it possible, throughout production, for the real development in the cross-linking process to be checked under the conditions prevailing at any time, so that the moulding can be automatically controlled accordingly without keeping to a previously prepared fixed timetable.

These results are achieved by the present invention, which provides an apparatus for checking or controlling the progress of cross-linking in elastomer products comprising a mould, carried by controllable closing means, consisting of two detachable parts defining an impression corresponding to the shape of one or more items made out of elastomer, the mould being able to withstand a cross-linking process that takes place inside it and being equipped with at least one pressure sensor rigidly mounted on one of the two parts of th mould, the sensitive surface of the sensor being in contact with the elastomer material undergoing cross-linking and being connected to the means for recording and/or processing changes in pressure within the impression of the mould containing the elastomer to be moulded.

In particular, according to one form of embodiment, the apparatus for checking or controlling the progress of cross-linking in elastomer products comprises a mould carried by controllable closable means consisting of two detachable parts defining an impression and able to receive a constant quantity of material which matches the shape of an elastomer test item, the mould being equipped with temperature control means and having at least one pressure sensor rigidly mounted on one of the two parts of the mould, the sensitive surface of the sensor being in contact with the elastomer material undergoing cross-linking and being connected to the means for recording changes in pressure inside the impression in the mould containing the

elastomer.

One of the parts of the mould has a hollow impression whereas the other has a projecting element which can be inserted into the hollow impression, so that a seal can be achieved between the respective coupled-together side walls, there being defined within the hollow impression a space able to receive an elastomer test item, the mould being mounted on closing means enabling the two parts to be tightly closed and locked one against the other, the projecting element of one fitting inside the impression in the other and the end of the run position being maintained to the end of the test.

In another form of embodiment of the apparatus for checking or controlling the progress of cross-linking in elastomer products according to the invention, the locking means carrying the mould comprise a moulding press and the mould has one or more pressure sensors located at significant regions within the mould, which are used to detect pressure changes in the mould during moulding and cross-linking, and are connected to a control panel which is provided for an automatic control of the successive stages in the moulding operation. More specifically, the apparatus according to the invention can be used in the production of elastomer products by injection moulding, in which case the locking means carrying the mould comprise an injection moulding press equipped with means for closing and opening the mould, an injection assembly having an injection unit by which the elastomer material is fed into the mould and a device for causing the injection assembly to move towards and away from the mould.

In this case, one or more pressure sensors are provided in the mould within the impression region or regions corresponding to the product, within the feeding or connecting channels between such regions and/or within a separate impression region specifically provided for that purpose, the pressure sensor or sensors being connected, together with other parts used to check the moulding, to a control panel comprising a microprocessor unit which, upon detection of certain values, is able to control the duration of the successive injection stages, the maintenance of a certain pressure level in the mould and the cross-linking.

Alternatively, in order to enable different moulding technologies to be used, the closing means for the mould may include a compression moulding press, a transfer press or a mixed press, the mould being provided with one or more pressure sensors connected to a control microprocessor able to regulate the duration of the cross-linking stage in the mould and when the latter is to be opened.

The method according to this invention provides a control of the cross-linking progress in elastomer products within a mould, comprising the measuring of pressure inside a fixed volume chamber of a mould containing the elastomer material, control of the decrease in pressure through cross-linking and the detection of the stage at which the gradient of decrease in pressure approaches zero or is anyhow lower than a pre-established value.

The method according to the invention can be used for injection moulding of elastomer products and the cross-linking thereof within the mould and comprises, in a press for the injection moulding of the products, the detection of the pressure existing at one or more areas in the mould as material is injected in a fluid condition, stopping the injection when the injection members have completed th ir operation and the pressure within the mould is higher than a pre-determined value at any area where it is measured, keeping the injection assembly up to pressure against the mould until, upon the detection of a decrease in the pressure corresponding to a pre-established value, it is moved away; opening the mould upon detection such that the pressure in the mould is further decreased below a second pre-established value or upon a decrease in the gradient of pressure variation within the mould.

More details will become apparent from the following description, made with reference to the attached drawings, wherein

Fig. 1 is a diagram showing how the torqu varies as time progresses at constant temperature in a vulcanisation tester having an oscillating rotor;

Fig. 2 shows a vulcanisation tester int nd d for laboratory tests, according to the invention;

Fig. 3 is a diagram showing how pressure varies as time progresses at a constant temperature when cross-linking is taking place;

Fig. 4 is a general diagram of an injection moulding press for an elastomer mixture wherein cross-linking is controlled according to the invention; and

Fig. 5 is a block diagram of the control apparatus for an injection moulding press.

The measurement of the progress of cross-linking in a specific elastomer mixture over a period of time and under constant temperature conditions is made, according to common practice, by means of a vulcanisation tester having an oscillating rotor, the vulcanisation tester being provided with a chamber filled with a fixed amount of th mixture to be measured and containing an oscillating rotor used to measur the torqu r quired to maintain an oscillation of constant amplitude during cross-linking.

The measurement shows an initial slight decrease of the torque caused by the homogeneous heating of the mass under test over a transitory stable lasting as a rule only a few minutes, followed

by a considerable increase of the torque when cross-linking is taking place, the torque regaining a practically constant value or only slightly varying as time progresses as a result of completion of the cross-linking process in the mass tested.

The progress of the test described above is shown, by way of example, in Fig. 1 which illustrates, for a sample of elastomer mixture, how a torque C (expressed in Nm) varies as a function of a time t (min) and shows an area 1 over which the torque has risen considerably, in correspondence to the stage of cross-linkage of the mixture, followed by an area 2 over which the torque is practically unchanged, once cross-linking is completed.

A study of how cross-linking of the mixture progresses may be carried out in an easier way by using the measuring apparatus according to the invention in the version for laboratory tests as shown in Fig. 2: it substantially comprises a mould made up of two halves, a male part 3 and a female part 4 defining a cavity 5, able to be sealingly locked together, within which cross-linking of the sample 6 of the elastomer mixture to be measured takes place.

The two halves 3 and 4 are provided with temperature control probes 7 and are mounted on a closure member, such as a press for instance, those initial compression force is kept unchanged throughout the test.

In one of the two halves 3 and 4 of the mould, in fact in the male half 3 in the example given, there is provided a hole 8 within which there is inserted a pressure sensor 9 whose sensitive tip 10, which is in contact with the test piece 6, is connected to a detection and control system comprising a recorder or a microprocessor unit able to produce a curved line showing how pressure varies inside the mould as time progresses and possibly to calculate and to provide directly the characteristic cross-linking parameters indicated by the curve.

The temperature control probes 7 must guarantee to a high degree of precision that the temperature of the sample be accurately maintained (suitably to a level lower than 0.3°C), so as to avoid expansions or contractions of the test piece caused by temperature variations throughout the test, which may considerably modify the pressure within the mould, resulting in further additions to the pressure variations caused by the cross-linking process.

A result of a cross-linking test carried out by means of the apparatus of Fig. 2 is shown in Fig. 3 illustrating the way a pressure P(MPa) varies as a function of a cross-linkage time t (min). As can be observed, over an initial region 12, the pressure increases after the press is closed until it reaches the value set for the test, there then follows a stage 13 during which the pressure decreases considerably until, over a region 14, it keeps to values which do not vary, or vary very little as time goes by.

The pressure decreasing stage 13 corresponds to the cross-linking of the tested mixture and it is possible to notice full agreement between the test carried out by means of the vulcanisation tester having an oscillating rotor (Fig. 1) and that carried out by measuring the pressure (Fig. 2); therefore, a measurement of the cross-linkage times taken by using the apparatus according to the invention appears to be fully able to provide operating, technological and theoretical directions, similar to those obtained by means of a vulcanisation tester having an oscillating rotor.

The mould used for the cross-linkage tests must be such as to guarantee that for the whole of the test the chamber is able to hold the same amount of material; to this end the male half of the mould has a plunger 15, aptly cylindrical in shape, suitable to fit with precision inside the cylindrical cavity 16 of the mould half 4, providing a seal between the matching cylindrical walls of the plunger and of the cavity, there being no surfaces in stepped contact, thus preventing possible side leakage of the material not yet vulcanised: the volume of the sample under test and in particular its thickness is therefore defined by the amount of material put inside the mould. A control stops the advance of the press part carrying the movable half of the mould when the pressure reaches the valu set for the beginning of the test as measured by the sensor 9: in order not to affect measurement of the pressure pattern within the mould, the type of press used, or the mould closure part, is of a type suitable to keep the position reached by the moving mould half constant throughout the duration of the test, so as not to cause variations in the volume of the test piece. In fact as the possibility of compression of the measured elastomers is very small, a small variation in the volume of the mould chamber would give unreliable pressure readings.

A suitable dimension for the test piece 6 may be for instance ~ 20mm diameter with a thickness of ~ 1mm.

A consistent thickness of the test piece 6 is dependent upon an accurate measurement of the amount of material introduced in the mould and it is therefore advisable for such measurement to vary in the order of ±10%: however, a variation in the thickness of the sample, proportional to the precision with which the material introduced is measured, has only a limited consequence on the duration of the transitory period over which the temperature of the mass of the sample reaches a consistent value: in fact, given the small size of the sample, whose thickness is suitably about 1 mm,

this transitory period is very short and therefore the effects of small variations in the thickness of the sample on the results of the measurement carried out are negligible.

It is also possible to provide a mould having bigger test pieces, and in this case a certain period of transitory temperature conditions is allowed during the test in order to obtain, after cross-linking, parts big enough to withstand further tests such as traction, hardness, resiliency, etc.

The apparatus and method according to the invention find application not only in the laboratory but also for controlling the operating cycle of an industrial press for moulding elastomer mixtures using all known moulding methods: injection, compression, transfer and a mixture of all these.

By way of example a basic injection moulding apparatus for elastomers is shown in Figure 4.

It substantially comprises a mould 17 including, in a simplified form, two mould halves 17A and 17B, carrying the impressions 18 of the pieces to be moulded, a mould closing assembly 19 carrying a movable part consisting, for instance, of a piston 20, or a toggle joint or other system acting on the half mould 17A, an injection part 21 provided with an injection actuator 22 and with an actuator 23 for causing the injector to move towards and away from the mould.

The press is also associated with a control panel 24 carrying the controls for adjusting the length of the cycle, the temperature and the operating pressure, possibly equipped with a microprocessor 25.

One of the impressions 18 of the mould 17, or other impression specifically provided for this purpose, depending on the form of the mould and of the pieces to be obtained, is equipped with a pressure sensor 26 connected to the control panel 24 of the press: the sensor 26 may be connected to an independent microprocessor or , when one is already present, to the same existing microprocessor controlling the press.

The diagram of Figure 5 shows how, in the case of injection moulding, the microprocessor is connected to detection parts which may comprise a quantity sensor 27, for instance an end of the run sensor able to stop the advancement of the injection assembly by means of the actuator 23, and several pressure sensors 26 suitably arranged within the impressions of the mould to detect pressure in the most significant areas, the microprocessor being also connected to actuators, comprising an actuator 23 for moving the injection assembly 21 towards and away from the mould, an actuator 20 for closing the mould and an actuator 22 for injecting the un-vulcanised material in a fluid condition into the mould.

The injection moulding cycle is controlled automatically by the microprocessor receiving signals from the sensor or sensors detecting the pressure and the position of the injection assembly and comprising the successive stages of closing the mould, approaching the injection assembly to the mould, injecting the material into the mould, keeping a certain pressure in the mould, the injection assembly being kept under pressure against the mould to prevent the material which has not yet undergone cross-linking from flowing back through the feeding channels, causing cross-linking of the item formed within the mould once the injection assembly has been moved away from the mould or no longer kept under pressure against it, and finally opening the mould after the item has undergone cross-linking.

A control ends the injection operation when the sensor or sensors detect that the pressure within the mould has reached a pre-established value and the correct amount of material has been introduced (corresponding to a position A of the diagram in Figure 3).

The subsequent stage, at which pressure is kept constant, is delayed until the pressure sensors indicate that cross-linking has begun or has progressed to a certain point (corresponding to a position B in Figure 3).

The cross-linking stage is therefore delay d until the pressure sensors indicate a decrease in pressure by a pre-established amount corresponding to the required degree of cross-linking (position C in Figure 3).

In this way, rather than working according to a fixed timetable for each stage as is the case in traditional moulding, the data collected throughout the experimental stage is used to control the length of the stages depending on realistic assessments of the conditions of the material within the mould.

It is therefore possible to avoid the drawbacks of conventional moulding which causes wastage and low productivity, such as a nil pressure in the mould caused by bad flowing of the material within the mould or by flowing back of such material after the injection assembly has been moved away, pre-cross-linking (scolding) in the injection nozzle and, chiefly, opening of the mould before the material is fully cross-linked or when it is over cross-linked: in fact, the apparatus according to the invention makes it possible, for each moulding operation, to detect the exact length of each stage, even when variations in the external moulding conditions occur, such as for instance temperature variations and the like.

The presence of one or more pressure sensors in the mould may be particularly advantageous in those cases, for instance when complicated moulds are used when there is fear that different areas of the mould may react differently: in such cases the

length of the successive stages is controlled by a verification that all the sensors show the expected readings in order to avoid the occurrence of areas of the mould where cross-linking is not completed. In moulds of certain shapes, it is also possible to use the signals from some sensors for controlling one stage, for instance the duration of the injection stage, by placing sensors in the most difficult areas to fill; the signals from other sensors may be used to control the length of the stage at which pressure is kept constant, for instance by placing sensors inside the feeding channels, whereas different sensors may provide a more correct indication of the completion of cross-linking by being located in areas where cross-linking occurs later.

In the case of compression moulding, transfer moulding or mixed moulding, for all of which the mould filling stage is comparatively easier than in the previous case, there are provided, within the mould, one or more pressure sensors connected to a controlling microprocessor, such sensors being used to control the progress of cross-linking within the mould with time and to control the opening of the mould when the pressure inside it has dropped to a pre-established value.

In this case too there is the possibility of carrying out an automatic control of the effective progress of cross-linking within the mould and therefore opening the mould only when cross-linking has reached the required stage, under realistic manufacturing conditions.

Many variations may be introduced without departing from the scope of the invention in its basic features.

Claims

1. Apparatus for use in controlling the progress of cross-linking in products made from an elastomer material within a mould, characterised in that it comprises a mould carried by controllable closing means and made up of two separate parts defining an impression corresponding to the shape of one or more pieces in an elastomer material to be subjected to cross-linking treatment within the mould; there being provided at least one pressure sensor rigidly mounted on one of the parts of the mould whose sensitive surface is in contact with the elastomer material undergoing cross-linking and is connected to means for recording and/or processing the variations of pressure within th impression in the mould containing the elastomer material throughout the moulding operation.

2. Apparatus for use in controlling the progress of cross-linking in products made from an elastomer material within a mould according to Claim 1, characterised in that it comprises a mould carried by controllable closing means and including two separate parts having an impression matching the corresponding shape of a sample of the elastomer material, such mould having temperature control means and there being provided at least one pressure sensor rigidly mounted on one of the parts of the mould, its sensitive surface being in contact with the elastomer material undergoing cross-linking and connected to means recording the variations of pressure within the mould impression containing the elastomer material.

3. Apparatus for use in controlling the progress of cross-linking in products made from an elastomer material within a mould according to Claim 2, characterised in that one of the parts of the mould has a concave impression whereas the other has a projecting part arranged to be inserted into the concave impression in such a way that a seal is provided between the matching side walls, respectively and a space suitable for receiving a sample of elastomer material is defined within the impression, the mould being mounted on closing means able to lock the parts against one another once the projecting element of the one part is inserted into the impression in the other part, the end of the run position being maintained constant throughout the test.

4. Apparatus for use in controlling the progress of cross-linking in products made from an elastomer material within a mould according to Claim 1, characterised in that the closing means carrying the mould include a moulding press and the mould has one or more pressure sensors located in the most significant areas of the mould impression in order to detect the pressure pattern during moulding and the progress of cross-linking within the mould, such sensors being connected to a control panel for the automatic control of the successive stages of moulding.

5. Apparatus for use in controlling the progress of cross-linking in products made from an elastomer material within a mould according to Claim 4, characterised in' that the closing means carrying the mould includes an injection moulding press equipped with means for locking and opening the press, an injection assembly having an injection unit able to feed the elastomer material into the mould and a member for causing the injection assembly to move towards and away from the mould.

6. Apparatus for use in controlling the progress of cross-linking in, products made from an elastomer material within a mould according to Claim 5, characterised in that one or more pressure sensors are provided within the mould in the impression region or regions corresponding to the product, and/or in the impression f eding or connecting channels and/or in a separate impression

region provided in the mould for this specific purpose, the sensor or sensors being connected together with the other mould controls to a display panel comprising a microprocessor able to control, according to the detected values, the duration of the successive injection stages, a constant pressure in the mould and the cross-linking.

7. Apparatus for use in controlling the progress of cross-linking in products made from an elastomer material within a mould according to Claim 4, characterised in that the closing means for the mould comprise a compression, a transfer or a mixed moulding press, the mould being equiped with one or more pressure sensors connected to a controlling microprocessor able to control the duration of the curing stage within the mould and to indicate the time when the mould should be opened.

8. A method for checking the progress of cross-linking in products made from an elastomer material within a mould, characterised in that it comprises measuring the pressure inside a mould chamber, which provides a fixed space, containing the elastomer material, checking the reduction of pressure during cross-linking, and detecting the stage at which the reduction in pressure approaches zero, or is below a pre-established value.

9. A method for use in injection moulding, and cross-linking inside a mould, products made from an elastomer material, characterised in that it comprises, in a press for injection moulding the products, measuring the pressure at one or more regions in the mould when the material is injected in a fluid state and interrupting the injection, when the injecting members have reached the end of their travel and the pressure within the mould, at all the spots where it is measured, is higher than a pre-established value, keeping the injection assembly under pressure against the mould, until it is moved away at the moment when a pressure drop by a first pre-determined value is noticed at one or more areas of the mould, and opening the mould upon verification that the pressure has dropped in the mould by a value higher than a second predetermined level or upon detection of a decrease in the gradient of pressure variation in the mould.

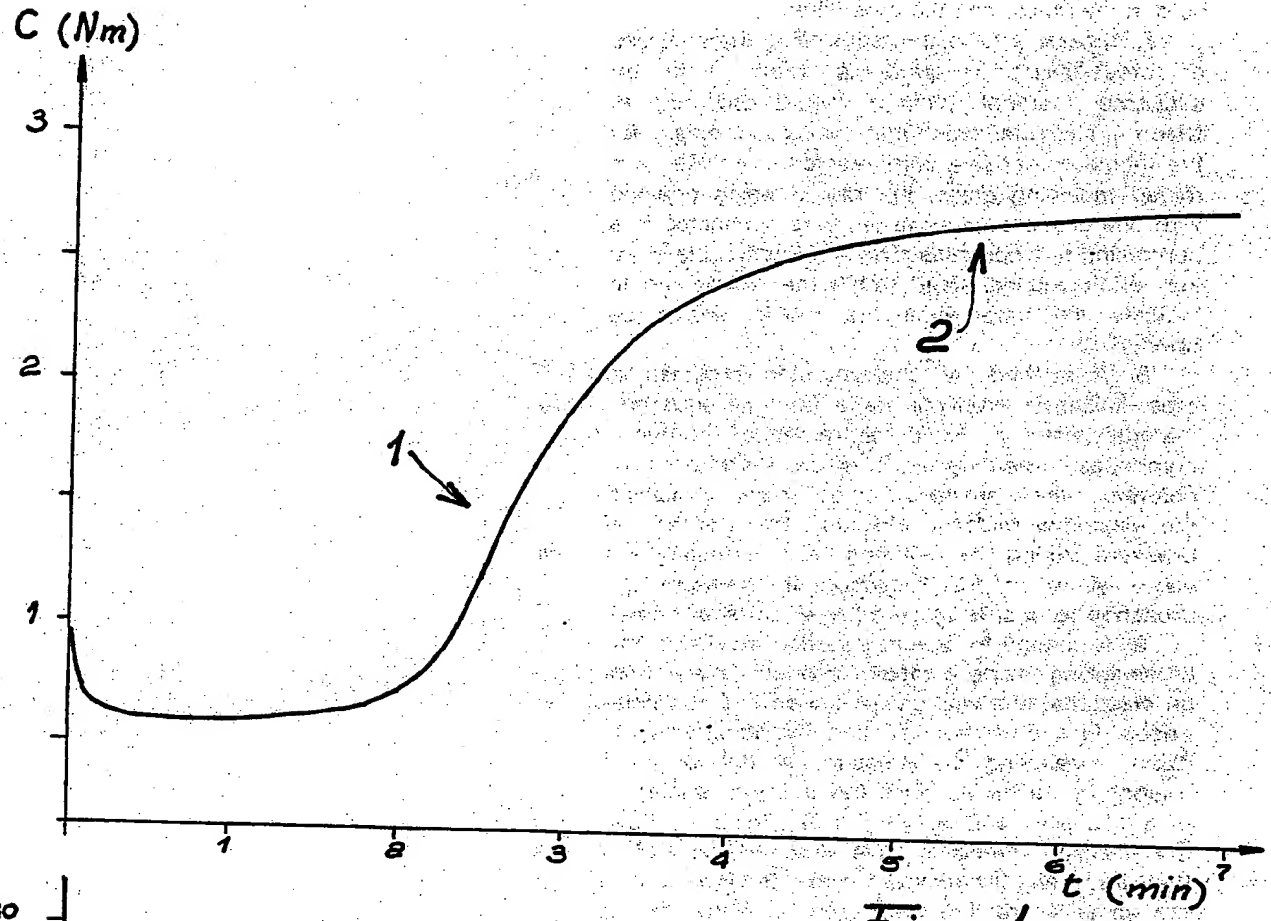

Fig. 1

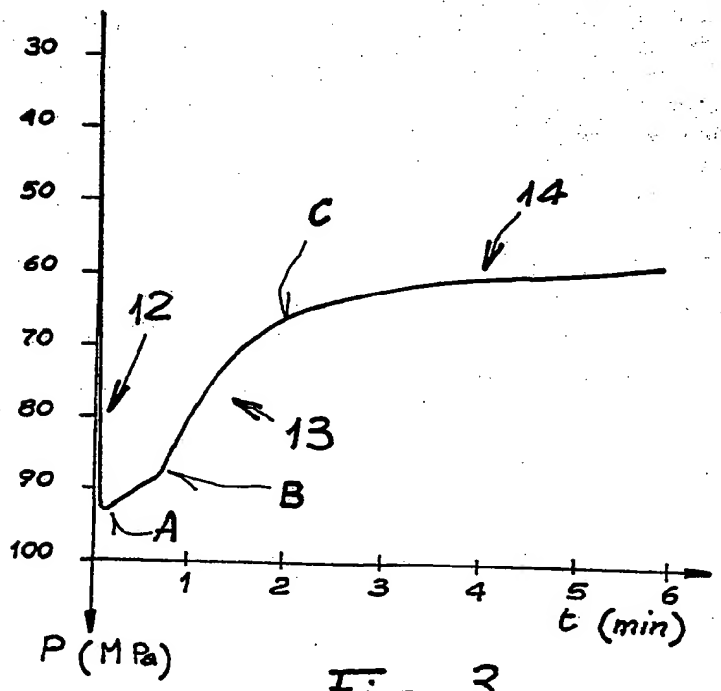

Fig. 3

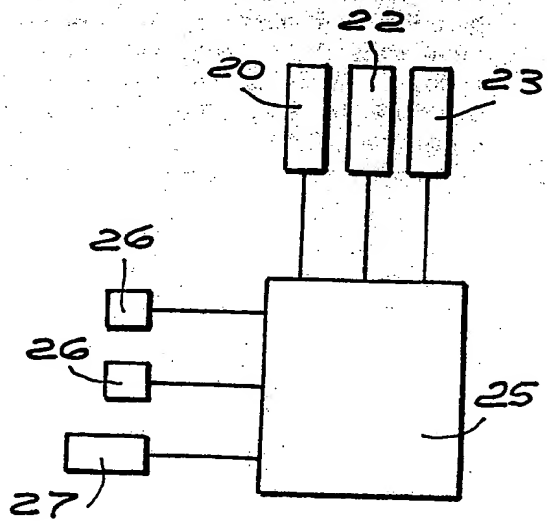

Fig. 5

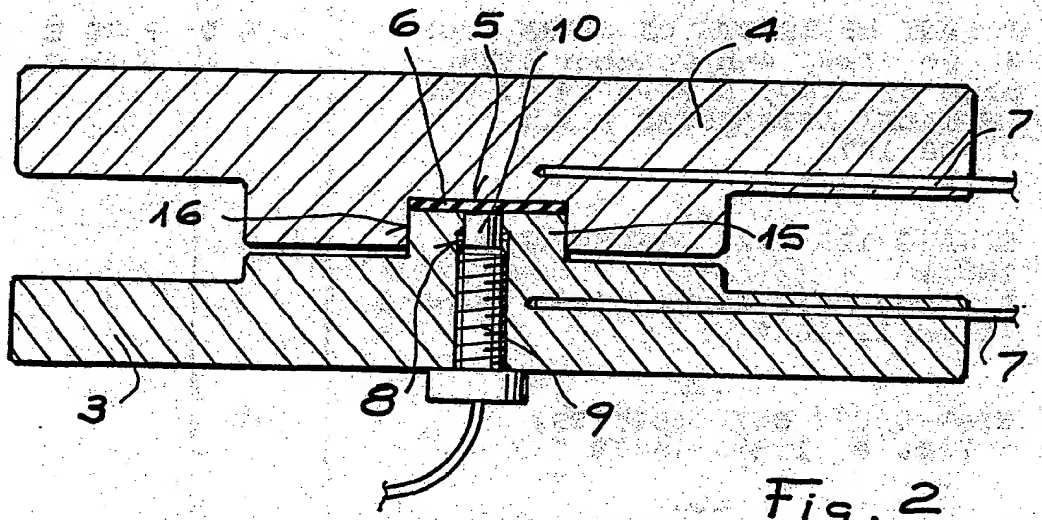

Fig. 2

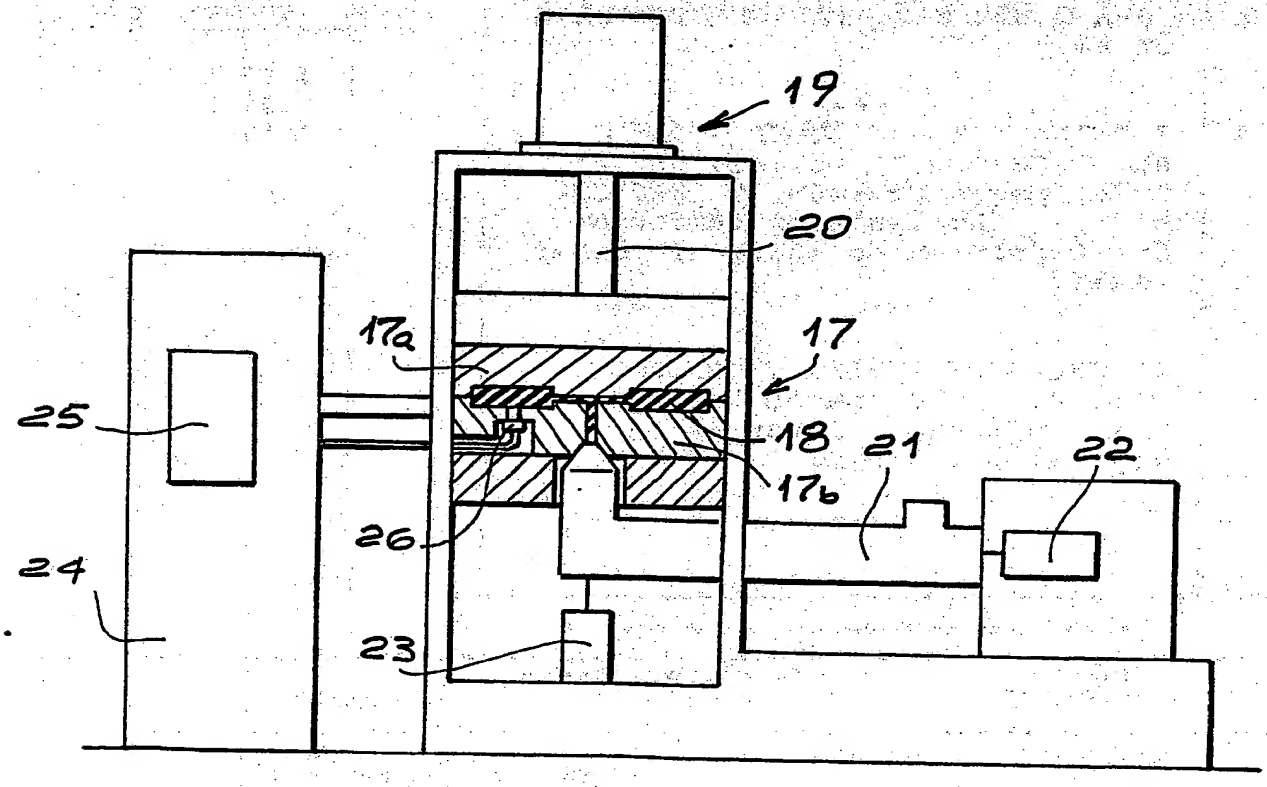

Fig. 4

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EUROPEAN SEARCH REPORT

Application number

EP 87 20 0238

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.4)
X	PATENT ABSTRACTS OF JAPAN, vol. 3, no. 118, 4th October 1979, page 52 C 60; & JP-A-54 97 660 (HITACHI SEISAKUSHO K.K.) 01-08-1979	1,8,9	B 29 C 45/77 B 29 C 35/02
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A	US-A-4 551 807 (HSICH et al.SE)	8,9	
A	US-A-3 929 012 (ANAGNOSTOPOULOS et al.)		TECHNICAL FIELDS SEARCHED (Int. Cl.4)
A	INTERNATIONAL POLYMER SCIENCE AND TECHNOLOGY, vol. 1, no. 2, 1974, pages T60-T63; M. ZAJICEK et al.: "Analysis of conditions for injection moulding of rubber mixes"		B 29 C B 29 H G 01 N
A	PROGRESS OF RUBBER TECHNOLOGY, vol. 42, 1979, pages 71-129; M.A. WHEELANS: "Advances in injection moulding"		

-/-

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	20-10-1987	ROBERTS P.J.

CATEGORY OF CITED DOCUMENTS

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EUROPEAN SEARCH REPORT

Application number

EP 87 20 0238

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.4)
A	EP-A-0 135 655 (KLÖCKNER-WERKE)		

TECHNICAL FIELDS SEARCHED (Int. Cl.4)

The present search report has been drawn up for all claims

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